

Sampling Report for Parent Drum S855793

UNCLASSIFIED

Forensic Science Center

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Section 1

Parent Drum Sampling

1. Summary of Sampling Activity

This report outlines the sampling of the empty parent drum of the breached drum identified in Panel 7 of the Waste Isolation Pilot Plant (WIPP). The 55-gal parent drum identification number is S855793 and it was contained in the 85-gal overpack drum 69120. Armando Alcaraz, of Lawrence Livermore National Laboratory (LLNL), was present to support and observe sampling operations conducted at Los Alamos National Laboratory (LANL) by LANL personnel. An overview of the sampling process and controls is depicted in Figure 1.

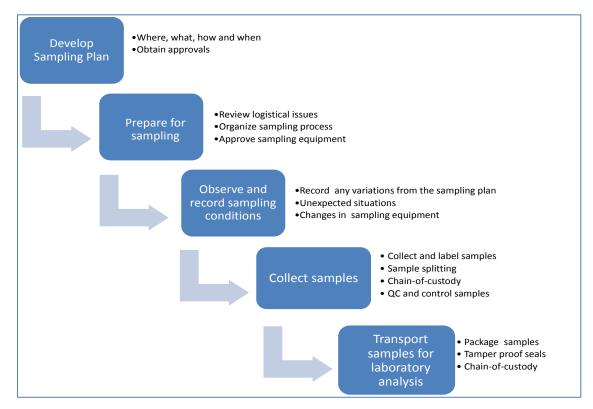


Figure 1. Sampling process flowchart

On Wednesday 8/6/2014 at approximately 1300, the headspace gases of drum 69120 were sampled at TA-54/Dome 33. Upon completion of the safety briefing and drum-handling

authorization paperwork, the composite overpack/Parent drum was moved into the LANL Drum Container Cabinet or venting and sampling/analysis of the headspace gases. This venting process involved the drilling and insertion of a special, long-shank drum filter and sampling port to penetrate the lid of the 85-gal overpack, as well as the lid of the Parent drum. The long shank provided direct sampling of the Parent drum headspace. Following placement of the long drum filter, a headspace sampler/transfer-line interface was attached to the filter and a sample of the Parent drum headspace gases (H₂, CO, CO₂, N₂O and volatile organic compounds) were collected and analyzed by an online gas chromatograph-mass spectrometer (GC/MS). When the GC/MS analyses were completed, the headspace sampler/transfer-line interface was removed and the overpack/Parent drum was moved to an open area inside Dome 33.

After on-line analyses were completed, additional headspace sampling was then performed using clean, 100-mL, gas-tight syringes. Two, 100-mL, gas samples were collected from the Parent drum through the long sampling port. Chain-of-custody (COC) forms were generated, and samples were placed inside a transport container. This activity completed the headspace gas sampling of the parent drum at TA-54/Dome 33.

The two headspace syringe samples were then transferred to TA-46, building 24. One sample was analyzed immediately, using gas chromatography with a thermal conductivity detector to detect permanent gases and by GC/MS to detect volatile organic compounds. The contents of the other gas-tight syringe were transferred to an evacuated 200-mL Summa canister for future TAT laboratory analysis (this transferred sample was at sub-ambient pressure). A TAT laboratory field blank was also collected in a 50-mL Summa (at ambient pressure) to establish the background CO₂ concentration for subtraction from the Parent-drum analysis (this blank was sent to SNRL for analysis).

On Thursday 8/7/2014, the 85-gal overpack/Parent drum was moved to TA-54 for denesting and sampling. The pre-job brief occurred from 0830-0915. Ann Schake (LANL), Patrick Martinez (LANL), Lisa Hudston (LANL), and Armando Alcaraz (LLNL) were present to support and observe the sampling operations.

The support team (Ann Schake, Patrick Martinez, Lisa Hudston, and Armando Alcaraz) arrived at 1000, at which point the drum operators and Radiological Control Technicians (RCTs) prepared the work area and the sample containers were staged. There were 4 Viking boxes readied for sample storage: SN 006 was designated for the "ARCHIVE" samples; SN 005 was designated for the "TATLAB" samples; SN 003 was designated for the "CMR" samples; and SN 007 was designated for the "TA-48" (radiochemistry site) sample.

A video camera was deployed to document the Parent drum denesting and sampling operations. It was staged immediately above the denesting apparatus such that it provided a very clear view of the focused work area. A video feed into the control room allowed remote observation by facility safety officers. During sampling operations, the drum operators and RCTs used powderless nitrile gloves specifically recommended for this activity by the TAT. The drum was brought into the contamination area (CA) at 1110. The operators denested the 55-gal Parent from the 85-gal overpack and prepared it for sampling. Patrick Martinez and the two samplers then proceeded to take all of the specimens that were outlined in the approved sampling memo

(Appendix C). When the Parent drum was opened, it was noted that no gasket material or free liquids were present.

Three different sample types were collected from the Parent drum for analysis: IAEA swipes for radiological analysis, glass swipes for organic analysis, and debris for targeted analysis and associated QC samples. Upon the completion of the Parent-drum collection process, samples were surveyed, their COCs were generated, and the samples then organized into their respective shipping boxes. A tamper-evident seal that had been provided by the samplers was applied to each sample. These seals are commercially available through a standard laboratory supplier (e.g., VWR).

The Parent drum was closed at 1245, and the drum operators exited the area. The support/observation team verified that the samples were in their respective shipping boxes, along with the attendant paperwork. The contents confirmed by Ann Schake, Patrick Martinez, Lisa Hudston, and Armando Alcaraz, and the samples were photographed by the TA-54 supervisory team. Specially-trained and certified LANL tamper-indicating device (TID) operators then applied LANL mylar TIDs to the shipping containers. Seal 037035L was applied to box 005 (TATLAB); seal 037036L to box 006 (ARCHIVE); seal 037037L to box 003 (CMR), and seal 037038L to box 007 (TA-48). The COCs were transferred to the TA-54 supervisor, and the team exited the area at 1415.

2. Sample Integrity

Samples were collected and placed within certified clean containers or their equivalent. The containers were secured with tamper-evident seals and were accompanied by COC documentation. The samples were stored in a secured area to prevent tampering or loss.

3. Quality Control Samples

Quality control (QC) samples were employed to verify sampling integrity and to detect contamination should it occur due to containers, handling, and transportation. The following types of QC samples were incorporated into the overall process:

- Field blank: Consists of an identical primary container as that used to collect the authentic sample, which is opened in the field for a similar period of time as required to take a sample, sealed, and transported to the laboratory for analysis.
- Transport blank: Consists of a new, sealed primary sample container, which accompanies the authentics samples during transport to the laboratory for analysis.

4. Sample Transport and Storage

During sample transport and storage, the following procedures were followed to ensure that samples were not altered and were in a condition suitable for analysis at the laboratory. The following sample transport and storage procedures were followed:

- 1. Samples were appropriately packaged to avoid breakage and cross-contamination.
- 2. Sample degradation was minimized through appropriate storage (e.g., maintained at room temperature).
- 3. Sample containers were sealed with tamper-evident seals and accompanied by COC paperwork.

5. Summary of Samples Collected

Samples were taken in three groups: 1) Samples for TAT laboratory analysis, 2) Archive samples and 3) Samples requested by LANL for potential analysis at LANL. For each sample collection method, both a field blank and transport blank were obtained. Three different sample types were collected from the Parent drum for analysis: IAEA swipes for radiological analysis, glass swipes for organic analysis, and debris for targeted analysis. Additionally, two drum filters were collected. Table 1 lists the specific samples taken by their COC numbers and description.

Table 1. Collected samples.

| | | TAT Laboratory Samples |
|-----|-----------------|---|
| No. | COC ID | Description |
| 1 | 69120-TATLAB-A | IAEA swipe sample |
| 2 | 69120-TATLAB-B | Solid debris in Teflon bottle ~20-30g |
| 3 | 69120-TATLAB-C | Field blank, empty Teflon bottle opened in CA |
| 4 | 69120-TATLAB-D | Glass filter swipe |
| 5 | 69120-TATLAB-E | Field blank, glass filter opened in CA |
| 6 | 69120-TATLAB-F | Transport blank, glass filter swipe not exposed to CA |
| 7 | 69120-TATLAB-G | Transport blank, Teflon bottle not exposed to CA |
| 8 | 69120-TATLAB-H | Field blank, IAEA swipe opened in CA |
| | | |
| | | TAT Archive Samples |
| 1 | 69120-ARCHIVE-A | IAEA swipe sample |
| 2 | 69120-ARCHIVE-B | Solid debris in Teflon bottle ~20-30g |
| 3 | 69120-ARCHIVE-C | Field blank, empty Teflon bottle opened in CA |
| 4 | 69120-ARCHIVE-D | Glass filter swipe |
| 5 | 69120-ARCHIVE-E | Field blank, glass filter opened in CA |
| 6 | 69120-ARCHIVE-F | Transport blank, glass filter swipe not exposed to CA |
| 7 | 69120-ARCHIVE-G | Transport blank, Teflon bottle not exposed to CA |
| 8 | 69120-ARCHIVE-H | Field blank, IAEA swipe opened in CA |
| | | |
| | | LANL Laboratory Samples |
| 1 | 69120-48 | IAEA swipe sample |
| 2 | 69120-CMR-1-B | Solid debris in Teflon bottle ~20-30g |
| 3 | 69120-CMR-1-C | Field blank, empty Teflon bottle opened in CA |
| 4 | 69120-CMR-2-A | Drum Filter Small |
| 5 | 69120-CMR-2-A1 | Drum Filter Long |
| 6 | 69120-CMR-2-D | Glass filter swipe |

6. Photographs of Sampling Activities

Appendix A contains representative photographs of various aspects of the sampling activity. Additionally, video of the sampling activity was taken and will be available in the TAT records.

7. Chain of Custody

Prior to this sampling activity Tamper Indicating Devices (TIDs) had been applied to the drums at LANL that were associated with the breached drum identified at WIPP. On Wednesday 8/6/2014, when the 85-gal overpack/Parent drum (S855793) was taken to the dome for venting/headspace sampling, photographs indicate the TID was in place during this process. On Thursday 8/7/2014, the 85-gal overpack/Parent drum was moved to TA-54 for denesting and sampling. At some point, either during or after the sampling activity, operators noted/observed they had not formally accounted for the TID on the drum. On Friday 8/8/2014, at 0845 the LANL Security Incident Team was notified and efforts were initiated to locate this missing TID. As of the date of this report, no additional information has been received by the TAT regarding this issue.

Appendix B contains copies of all COC forms at the time of sample collection.

8. Sampling Authorization

Sampling of the parent drum was approved by the TAT based on a sampling memo provided by LANL. A copy of the approved sampling memo is enclosed in Appendix C.

Appendix A

Photographs of Sampling Activity



Figure~2.~Overpack/Parent~drum~at~the~LANL~Drum~Container~Cabinet~(DCC)~for~venting~and~sampling/analysis~of~the~headspace~gasses.



Figure 3. Headspace syringe sample being transferred into a Summa canister for TAT laboratory analysis.



Figure 4. Removing the Parent drum from the overpack drum at TA 55.



Figure 5. View inside empty Parent drum.

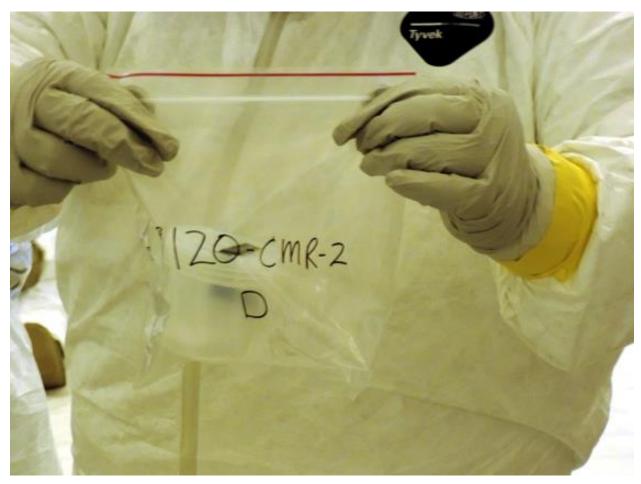


Figure 6. Sample 69120-CMR-2 D, Glass filter swipe being packaged inside hot area.



Figure 7. Samples being packaged inside Viking containers.

Appendix B

Chain of Custody Forms for Samples Collected

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| 69120-7AT LAB A | TA | IAEA Swipe sample | | |
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Appendix C

TAT Approved Sampling Memo



memorandum

Actinide Analytical Chemistry

To/MS: Nancy Sauer, ADCLES, F629 *From/MS*: Rebecca Chamberlin, C-AAC, G740

Phone/Fax: 7-1841/Fax 5-4737 Symbol: C-AAC-14-0035 Date: August 1, 2014

Subject: Sampling Plan for Empty Parent Drum S855793 (69120)

LANL staff will sample empty drum S855793 (overpack 69120) in accordance with Procedure EP-AREAG-WO-DOP-1245, R.O. The following sample sets will be obtained, in accordance with guidance from the TAT Sampling Team, as provided by Armando Alcaraz, LLNL.

- 1. TAT Laboratory Samples (labelled "69120-TATLAB-#")
 - a. One (1) IAEA swipe sample.
 - b. One (1) sample of bulk solid debris, approximately 20-30 g.
 - One (1) field blank, using bulk solid sample container, opened in the sampling area as soon as the drum lid is opened.
 - d. Glass filter swipes, as needed (see discussion below).
 - e. Portion of drum gasket, if available (see discussion below).
 - f. Portion of free liquid, if available (see discussion below).
 - g. One transport blank per shipping container, not exposed to sampling area.
- 2. TAT Archive Samples (labelled "69120-ARCHIVE-#")
 - a. One (1) IAEA swipe sample.
 - b. One (1) sample of bulk solid debris, approximately 20-30~g.
 - c. One (1) field blank, using bulk solid sample container, opened in the sampling area as soon as the drum lid is opened.
 - d. Glass filter swipes, as needed.
 - e. Portion of drum gasket, if available.
 - f. Portion of free liquid, if available.
 - g. One transport blank per shipping container, not exposed to sampling area.
- 3. LANL Laboratory Samples (labelled as per EP-AREAG-FO-AP-1097)
 - a. Two (2) drum filters, if available.
 - b. One (1) IAEA swipe sample.
 - c. One (1) sample of bulk solid debris, approximately 20-30 g.
 - d. One (1) field blank, using bulk solid sample container, opened in the sampling area as soon as the drum lid is opened.
 - e. Glass filter swipes, as needed.
 - f. Portion of drum gasket, if available.
 - g. Portion of free liquid, if available.

General Information on Samples: Samples will be triply contained (*e.g.* vial + 2 ziploc bags; swipe + 3 ziploc bags). The final outer bag will be decontaminated to free-release levels and a self-adhesive TID will be placed on the outer bag. The innermost container is expected to have external contamination; intermediate containment layers may also have low levels of contamination.

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C-AAC-14-0034 - 2 - July 25, 2014

Bulk solid debris samples are expected to contain alpha-emitting radionuclides. 239 Pu is typically present in these samples at 10^{-5} Ci/g of debris, while 241 Am is typically present at 10^{-4} Ci/g of debris.

Bulk Solids Containers: LANL has procured wide-mouth short-profile jars, certified cleaned to EPA specifications, for this operation. Suitable HDPE and glass jars were available to ship in the appropriate timeframe.

Swipe samples: Samples will be taken for radionuclide analysis using IAEA environmental swipes. To improve contact with the drum surface, a laminated card will be held behind each swipe. The card will be deposited in the sample bag with the swipe.

Three sets of glass fiber swipes will be available if visual observation of the drum suggests unique value in these additional samples. For example: (1) if free liquid or oily residue is observed, (2) if the "debris tail" created when the drum was emptied has a distinct visual appearance from the bulk material at the bottom of the drum, (3) to collect the outer surface of a sticky solid residue. The chemistry SME will make this determination after the drum is opened. Any such samples should be obtained in triplicate for the three sample sets.

Drum gasket: Based on past sampling of empty nitrate salt parent drums, LANL does not anticipate that the drum gasket will be present. However, if present, the gasket will be sectioned for each sample set using cutting pliers with pre-cleaned cutting tips (cleaned with methylene chloride and isopropanol).

Free liquids: Based on past sampling of empty nitrate salt parent drums, LANL does not anticipate that any free liquids will be present. However, if present, the liquids will be sampled with pre-cleaned disposable plastic pipets.

Decontamination Procedures: When radiological decontamination is needed, personnel will avoid the use of Fantastik to the extent practicable. Deionized or HPLC-grade water will be supplied and should be used first for decontamination of surfaces. Tape-lift methods are also acceptable. Fantastik should be used only if radcon objectives cannot be met by the other methods.

Glove changes: Use of scented talc (baby powder) between glove layers is to be avoided, if possible. If the use of talc cannot be avoided, donning and doffing should occur away from the drum opening and near the floor, to prevent contamination of the samples.

Chain of Custody: LANL Chain-of-Custody procedure (QP-5.8, R.0) and forms will be used during sample collection and shipping. Tamper Indicating Devices (TIDs) will be placed on each sample's outermost layer of containment for added security (e.g., an adhesive tamper evident seal with unique numbers and/or letters). Receiving labs may implement their own internal chain-of-custody procedures once samples are received and accepted.

Shipping: Shipping containers (Viking boxes) will have a TID applied. The locations for the TAT Laboratory and TAT Archive sample sets had not been identified at the time of this memo. Area G personnel will store the TAT samples in a locked area until shipping occurs.

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